Monday, 8 June 1998

#### 1998 IMS Workshop WMG:

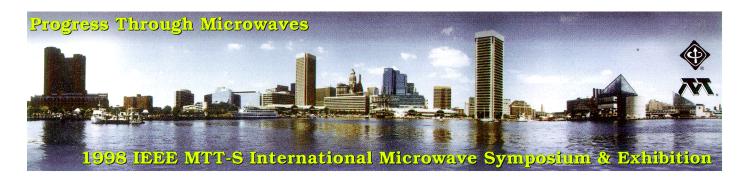
**Cryogenics: A New Beginning** 

# Setting Up and Calibration of a Cryogenic Test Station

J. M. Pond

Code 6851, Naval Research Laboratory Washington, DC 20375

pond@chrisco.nrl.navy.mil







#### **Outline:**

- Introduction
- Overview of a system
- The cryogenic refrigerator
- The vacuum chamber
- Providing a vacuum to insulate the experiment
- Temperature monitoring and control
- Getting an isothermal environment
- Getting microwave signal in and out
- Calibration issues
- Some references and conclusions



#### **Motivation:**

- Provide some useful information to rf and microwave engineers who:
  - have little or no experience with cryogenics
  - have little or no experience with vacuum equipment
  - wish to save themselves some grief by finding out from someone who has been greatly grieved what does and doesn't work
- Initiate and stimulate discussion of techniques for improving cryogenic microwave measurements and testing methods
- Firm belief that, even if HTS hasn't "lived up" to the popular hype, cryogenic electronics is a near term reality:
  - colder is better higher conductivity, higher mobility, less noise
  - cryocooler and packaging technology is becoming affordable

#### **Disclaimers:**

 Where possible information about vendors and suppliers has been provided. In no case should this be presumed to be an exhaustive list nor a recommendation or endorsement



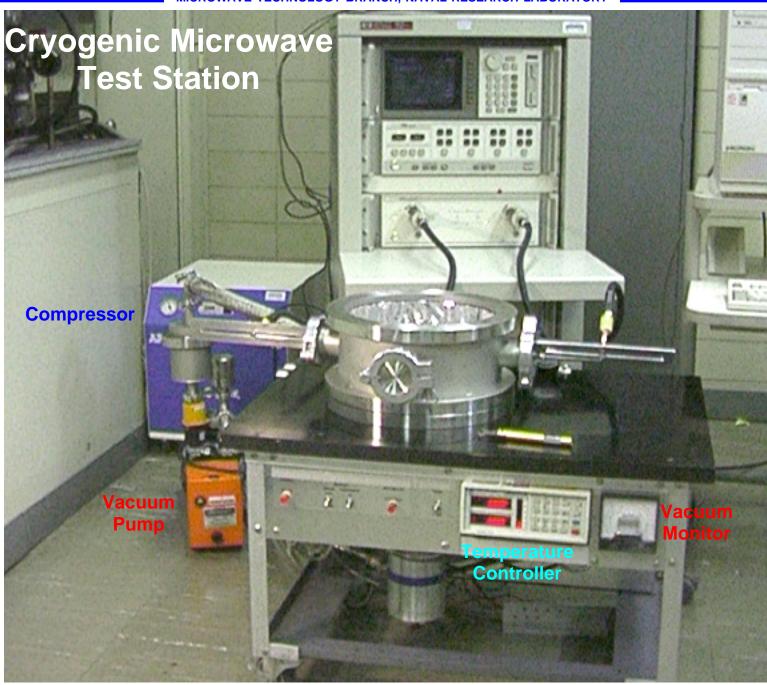
#### **System Options:**

- Closed-cycle refrigerator
  - Vibration can be a problem microphonics
  - Self contained no transfer of cryogenic fluids no cryogenic infrastructure required
  - Reliable based on cryopumps found in vacuum systems
  - Not cheap
  - Ensuring an isothermal environment is more difficult
- Open cycle need cryogenic fluid (liquid Nitrogen or Helium most common)
  - Nitrogen is cheap and easy to handle but can't get much below
     77K
  - Good if you can immerse in LN and are happy with one temperature and can stand condensation on device
  - Liquid Helium requires much more expertise in handling can get below 4K but is more expensive
  - With either, if you want temperature control over wide range you approach complexity of closed cycle system



#### **Components of a Closed Cycle System:**

- Refrigerator
  - Compressor where heat is expelled air or water cooled
  - Expander the gas expands and cools the "cold finger"
  - Gas lines often the expander and compressor are separate units with two Helium gas lines connecting them (He plays the role that Freon plays in your household refrigerator)
- Vacuum system vacuum provides the insulation that allows the DUT to be cooled
  - Pump various options from roughing pumps to turbo pumps
  - Gauge allows monitoring of chamber pressure
- Temperature regulation refrigerator runs unregulated heaters provide ability to adjust temperature
  - Heaters resistors
  - Sensors (thermometers) many options
  - Controller feedback loop between heater and sensor
- Vacuum chamber provides thermal isolation of DUT
  - Feed throughs electrical, mechanical, optical





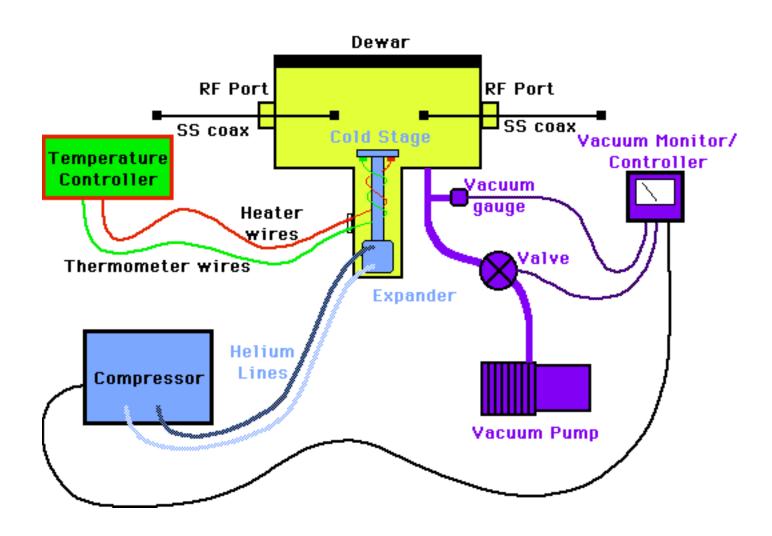
### The Chamber, Microwave Feedthrough and Temperature Controller





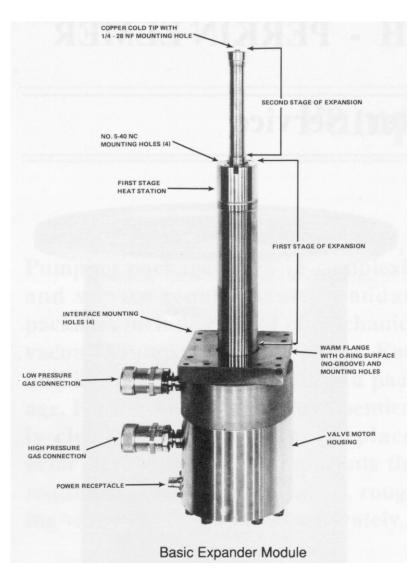


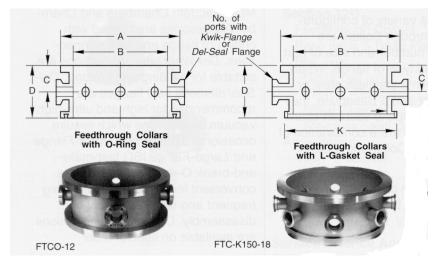
## The Basic Subsystems of a General Purpose Cryogenic Microwave Test System



#### **Some of the Basic Components**







**Basic Vacuum Chamber** 



Viewport/Optical



**Sliding Coax Feedthrough** 



#### **Laboratory Closed-cycle Refrigerator:**

- Assuming size, weight, and power consumption are of minimal importance in laboratory setting, need to estimate:
  - Minimum operating temperature
  - Maximum heat load at that temperature
    - generated within the DUT/sample
    - due to thermal radiation and thermal conduction
- Many closed-cycle systems for laboratory use have been developed from the cryopump industry:
  - Usually Gifford-McMahon cycle
  - Typical cooling capacities: 10-50 Watts @ 77K and 5-15 Watts
     @ 20K
- Some vendors:
  - APD Cryogenics (www.apdcryogenics.com)
  - CTI-Cryogenics (www.ctivacuum.com/enhanced/index.htm)
  - CVI (www.chart-ind.com/cvi/crs/)
  - Leybold Cryogenics (www.leyboldcryogenics.com)

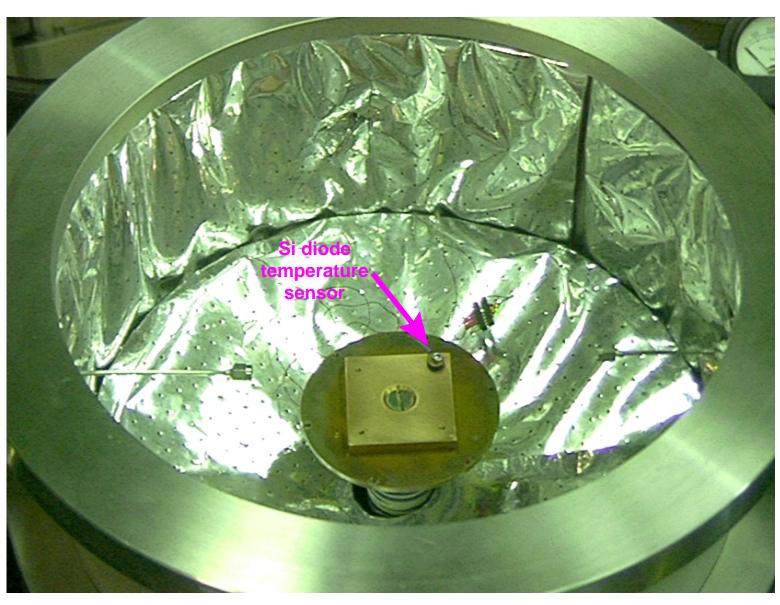


#### **Vacuum Chamber:**

- Rely on standard commercial parts as much as possible
  - Stainless steel components readily available
    - stainless is the standard for UHV minimal outgassing
    - stainless is heavy
    - wide range of standard component (building blocks) available to minimize custom work
  - Custom stainless steel vacuum equipment is expensive
    - welding to UHV standards is not trivial
    - brass, aluminum, lucite, etc. are fine for custom/home made parts
- Some vendors:
  - MDC Vacuum Products Corp. (www.mdc-vacuum.com)
  - Nor-Cal Products (www.snowcrest.net/norcal/index.html)

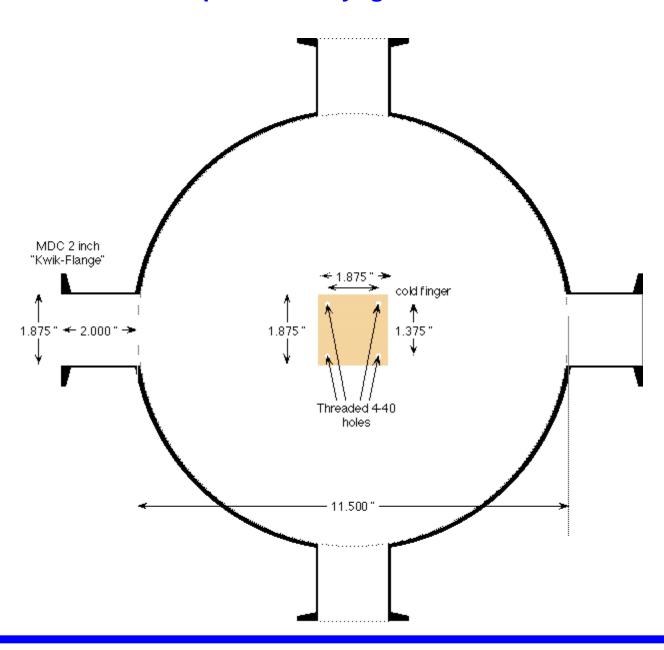
### Close-up of the Chamber, Cold Stage and Si Diode Sensor







#### **Top View of Cryogenic Chamber**

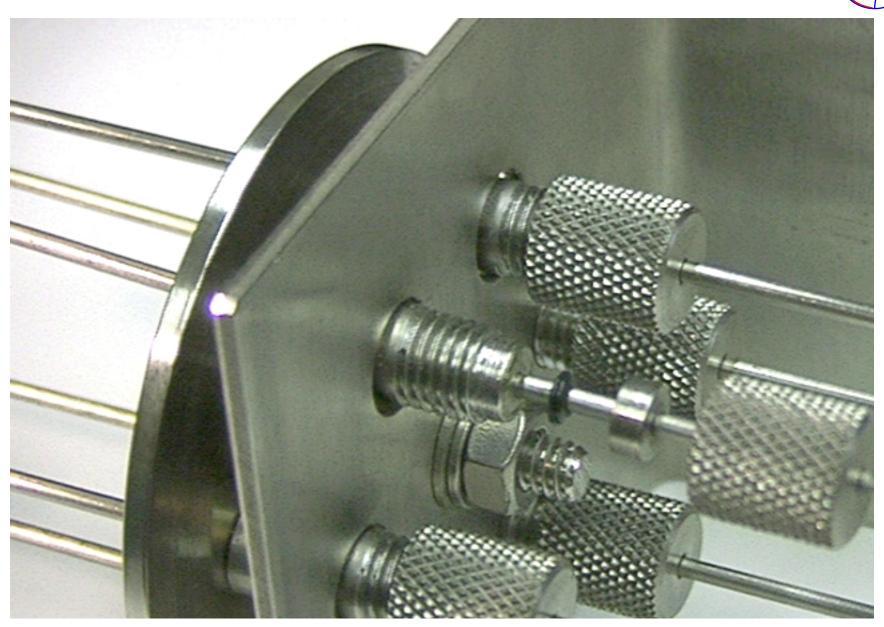




#### **Vacuum Chamber — Feedthroughs:**

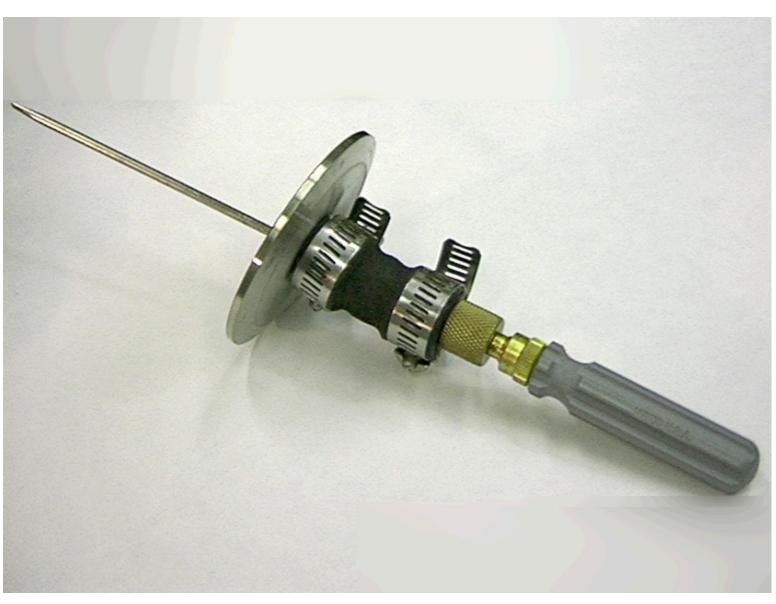
- The ports and feedthroughs are as important as the chamber itself
  - Flexibility
    - have plenty of standard ports using one (maybe 2) flanges
    - brass, aluminum, etc. are fine for custom parts
  - Customizable
    - standard flanges always have blank-off plates available
    - an excellent starting point for custom feedthroughs
  - Expandability you always need more feedthroughs
    - do not risk integrity of the basic chamber
    - your needs will grow and it is best to be able to keep the basic system geometry intact
- Types of feedthroughs (besides the obvious: coax cable and bias)
  - Mechanical
    - tuning screws on DUT, etc.
  - Optical
    - visual inspection troubleshooting show and tell
    - optical response
  - Radome

### **Close-up of 6-port Sliding Coax Feedthrough**



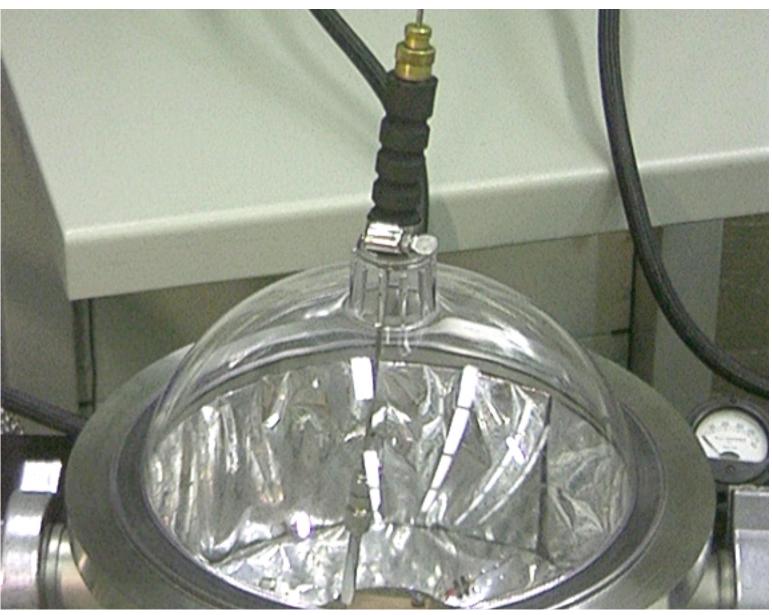
### **Simple Mechanical Feedthrough for Tuning DUT**





## A Plexiglass Desiccator Cover for a Radome or for Visual and Mechanical Access







#### **Vacuum Considerations:**

- With decent cooling capacity a rough pump down to ~100 milliTorr is sufficient. The refrigerator will then act as its own cryopump.
- Since the refrigerator is a better vacuum pump than a roughing pump, "backstreaming" of oil into the chamber can be a problem
  - oil can coat the chamber, DUT and cabling a mess at best
  - solutions:
    - valve off roughing pump automate by using a pressure gauge/controller (www.granville.com/275cam.htm, www.mdc-vacuum.com)
    - use turbo pump to evacuate chamber
- With a good roughing pump and large refrigerator, virtual leaks, outgassing and very small leaks are usually not a problem.
- Vent with Nitrogen to eliminate condensation on cold stage/DUT
- Some vacuum pump/system vendors:
  - Alcatel Balzers Edwards Leybold Sargent Welch Varian
    - Precision Shimadzu Seiko Seiki Kinney Stokes CTI

#### **Temperature Control**



- Most systems use a refrigerator running at full capacity and use resistive heaters in conjunction with thermometers and a controller to provide an isothermal testing environment.
- Standard practice is to use two thermometers
  - Temperature control mounted close to the heater
  - Sample temperature mounted on or as near as possible to the DUT/sample
  - \Delta T of thermometers is a good measure of thermal design
- To provide good control at all temperature:
  - Heater power > cooling power at all temperatures
  - Ohmite "Metal-Mite" aluminum housed axial lead wirewound resistors are a very robust heater (E.J. Cukauskas)
  - "PID" controller provides best performance
  - Autotuning features can be useful
- Cryogenic Temperature Controller manufacturers include
  - Lakeshore Cryotronics (www.lakeshore.com/)
  - Scientific Instruments, Inc. (www.sci-inst.com/)

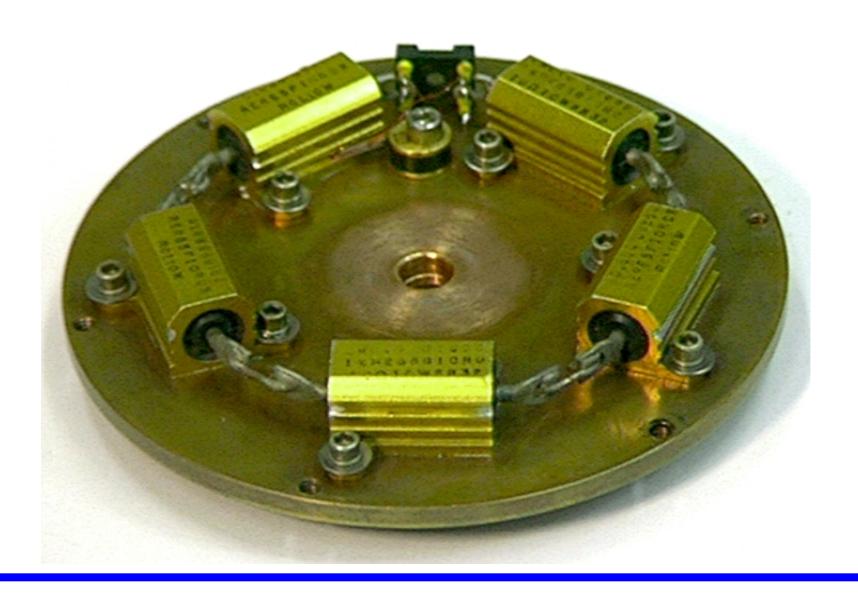


#### Thermometers/Sensors:

- Types:
  - Diode fast response, interchangeable, standard calibration
  - Resistor larger, mounting strain induced errors, many types with both PTC and NTC
  - Capacitor drift problems and thermal cycling problems, immune to magnetic fields
  - Thermocouple errors due to temperature gradient of wire
- Characteristics excellent product and Application Notes are available from vendors — make sure your choice meets your needs:
  - Magnetic field sensitivity
  - Size and Mass
  - Reproducibility and Stability
  - Accuracy/sensitivity/resolution
  - Interchangeability
  - Response time
- Recommendation:
   Unless **H** fields are part of your experiment use 4-wire Si diode

#### **The Heater Resistors and Control Sensors**

(This is the underside and mounts directly on the cold finger)



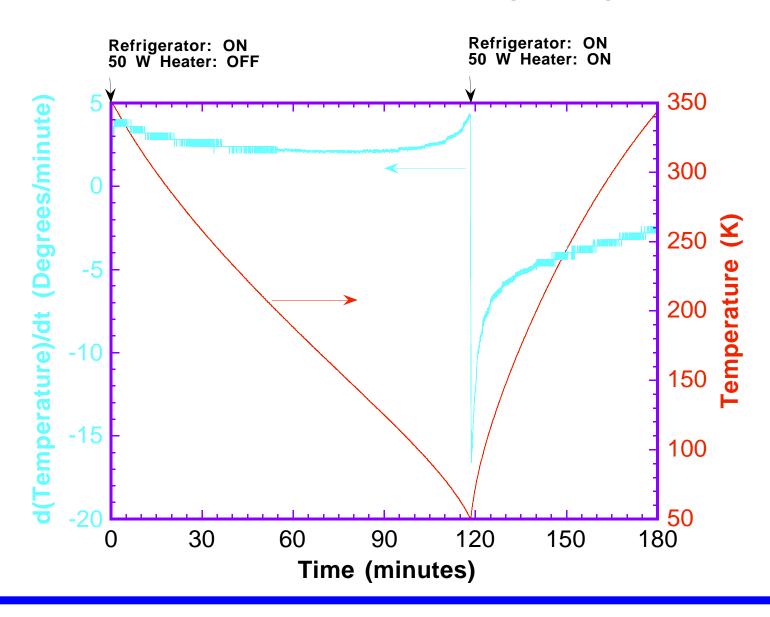


#### **Ensuring Good Thermal Contact:**

- Minimize number of interfaces that heat must cross.
- Utilize materials such as Oxygen Free High Conductivity (OFHC) copper in as much of the cold finger and DUT mounting platform as possible.
  - not easy to machine
  - particularly important for working much below 77K
  - www.copperandbrass.com
- In all cases it is preferable to directly mount (screws) or clamp the DUT/ sample to the cold stage rather than rely on gravity.
- Once the chamber is under vacuum, thermal contact of DUT/sample to cold stage must be ensured. Options include:
  - Thermal grease Apeizon Type N or equivalent messy
  - Rubber cement particularly good for small sample where concern about use of solvents exists
  - Indium foil more difficult to use may "stick" to sample and be destroyed in removing — expensive
  - Au foil/indium foil even more expensive Au is inert



## Thermal Behavior of Cryogenic Microwave Test Facility Temperature Versus Time and Cooling/Heating Rates





#### **RF Feedthroughs:**

- Balance the issues of attenuation versus heat leak.
- Heat leak not only loads the cooling capacity of the cooler it also is dumping heat directly into the region of the DUT/sample that is being measured leading to higher temperature uncertainty.
- In many laboratory cases, trading off attenuation for better thermal conditions is preferable the real world is much more challenging.
- Stainless steel coax is popular option compared to Cu

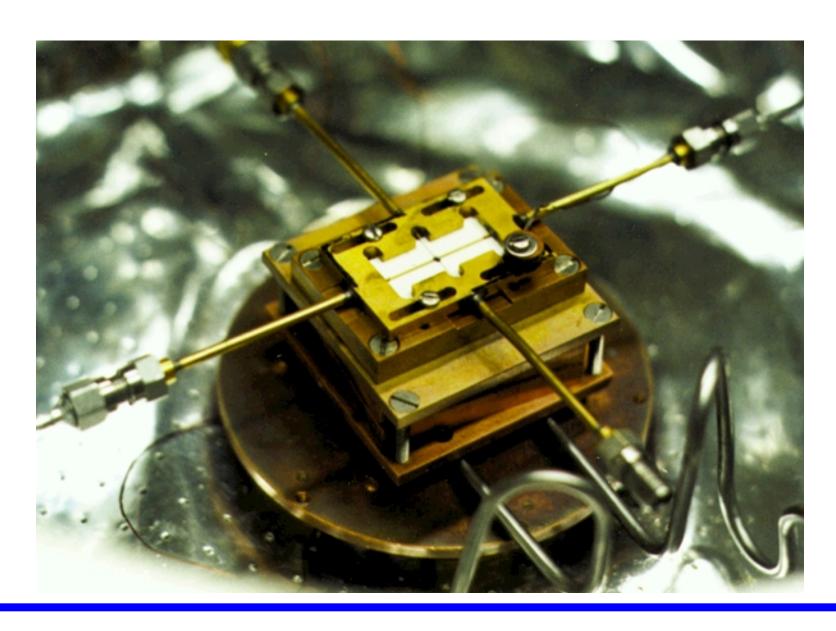
Cable Type	Thermal load (W•cm)	Insertion Loss (dB/cm)
0.085 Cu	14.97	0.022
0.141 SS	1.77	0.028
0.085 SS	0.90	0.048



#### **Coaxial Cable and Connector Considerations:**

- Teflon has high coefficient of thermal expansion
  - gaps can occur at low temperatures
  - if possible use coax with special dielectric with low coefficient of thermal expansion (ISOCORE by Rogers is no longer made)
- Hysteresis in teflon creep can lead to problems:
  - Center conductor contact problems "captured center pin" connectors may be required especially for long straight cables.
  - Measurement repeatability and, hence, calibration problems
- Any of the standard coax connector series (SMA, K, OS-50) can be used — individual connectors within a series may be suboptimal
- Stainless cables require stainless compatible solders and fluxes.
   Suggested solutions:
  - Eutector Flux 157 and EutecRod 157 (Eutectic Corporation, 40-40 172nd St., Flushing, New York 11358)
  - Indium Corporation of America (www.solder.com/index.html)

## **Cold Stage with DUT Mounted Background is 10 Layer MLI**





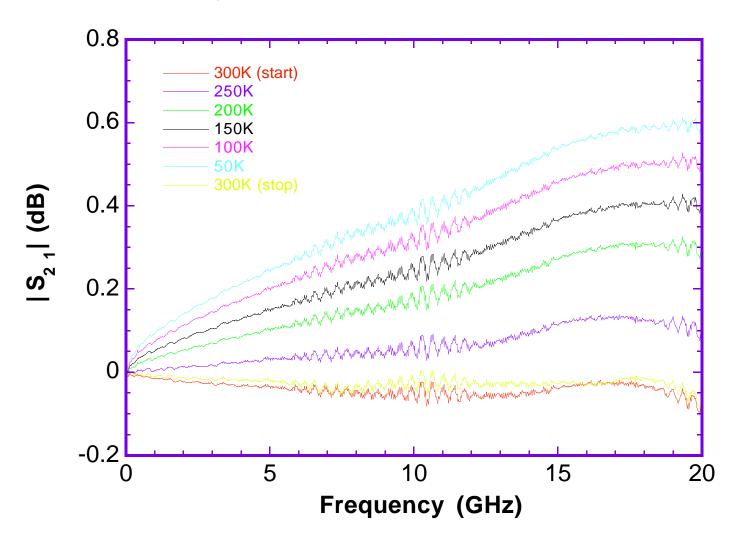
#### **Measurement and Calibration Issues:**

- Everything has some amount of temperature dependence measurement calibration is an issue
  - If nothing else, the physical length changes
  - IL of coax decreases electrical length decreases
- Room temperature calibration
  - reduced accuracy
  - easy same as always
  - some first order correction for IL can be made in some cases
- Cryogenic calibration
  - Standards, particularly loads, have temperature dependence themselves — TRL preferable
  - Tedious unless a matched network using switches is used, a cryogenic cycle is needed for each standard
  - Instrument drift may be an issue
- Cryogenic calibration
  - Standards, particularly loads, have temperature dependence themselves — TRL preferable



# Measurement Error at Various Temperatures Illustrated by Measurement of an SMA F-F Bulkhead Adapter After Room Temperature Calibration

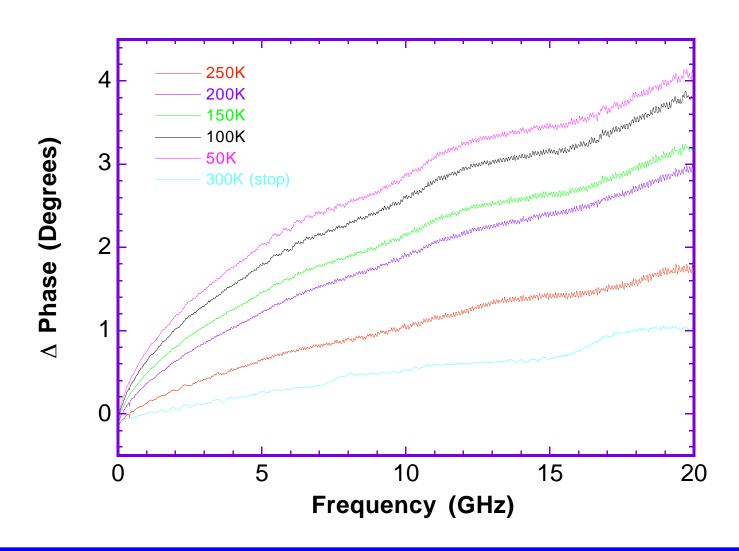
(using HP 85052D (SOL) 3.5mm economy cal kit)





# Measurement Error at Various Temperatures Illustrated by Measurement of an SMA F-F Bulkhead Adapter After Room Temperature Calibration

(using HP 85052D (SOL) 3.5mm economy cal kit)





#### **Measurement and Calibration Issues (cont.):**

- The previous example was a worst case in many ways:
  - The cable length subject to a thermal gradient was at a maximum (~20 cm)
    - IL can be reduced by ~2X by using 0.141
    - Use shorter cables 5 cm of 0.141 cable would have a 0.5 Watt thermal load but would reduce IL by ~4X
    - phase error would, similarly, be reduced by ~4X
  - Based on measurements shown in previous graphs, these modifications should result in IL error <~0.1 dB and phase error <~0.5 degrees</li>
- Possible complications:
  - Thermal design of DUT/sample becomes more of an issue since more heat is entering the DUT coaxial connectors.
    - minimize layers and interfaces in DUT package
    - place sample temperature sensor near coax connector
    - more sample temperature sensors to monitor ΔT of DUT
  - May need to customize coax feedthroughs to optimize this performance for each DUT



#### **Measurement and Calibration Issues (cont.):**

- Noise measurements can be particularly tricky to the unwary
  - Standard Noise Figure Meters:
    - assume that both ports of the DUT are available
    - are scalar instruments hence have problems with electrically long low-loss devices
    - cryogenic feedthroughs represent just such a problem additionally their electrical length and loss are temperature dependent
    - will happily report negative Noise Figures
  - Proceed carefully from first principles
    - even by deembedding from the cascade doesn't really solve the problem due to reflections
    - an isolator can help with the reflection problems and make deembedding more accurate in some cases
  - Listen carefully to the two talks in this Workshop on this important topic
    - low Noise Figure is one of the primary reasons cryogenic operation is so promising
    - it is the measurement area where standard commercial RT techniques are least applicable



#### **Some Suggested References:**

- Cryogenic Engineering, R.B. Scott, D. Van Nostrand Co. Inc., Princeton, New Jersey, 1959
- Experimental Techniques in Low-Temperature Physics, Third Edition, G.B. White, Oxford Science Publications, Clarendon Press, Oxford, 1979
- Cryogenic Process Engineering, K.D. Timmerhaus and T.M. Flynn, Plenum Press, New York, 1989
- Temperature Measurement and Control Catalog, Lakeshore Cryotronics, Inc., Westerville, Ohio
- Web sites:

www.omega.com/techref/itemp.html — The International Temperature Scale of 1990 (ITS-90)



#### **Conclusions:**

- Performance advantage of cryogenic operation is obvious.
- Cryocoolers for product are increasing in reliability and decreasing in price — particularly if LN temperatures are not needed.
- Cryocoolers for laboratory use are very rugged and reliable.
- Assembling a laboratory system is basically an exercise in assembling off-the-shelf hardware from different fields.
- Custom hardware can be minimized
- Measurement and calibration of a cryogenic system at microwave frequencies, although more difficult than at room temperature, involves basic trade-offs of speed versus accuracy — analogous to issues that affect room temperature measurements as well — just not as familiar to the microwave engineer and there is not, as yet, a solution offered by the major microwave test and measurement industry.